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Research Article

Micropore Structure Characteristics and Recoverability Evaluation of Typical Shale Oil Reservoirs

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In view of the weak research on the availability of typical shale oil reservoirs from the perspective of development, this study introduced a two-dimensional nuclear magnetic resonance (NMR) evaluation method on the basis of the previous one-dimensional NMR combined with centrifugal physical simulation experiments. Not only the production characteristics of typical shale oil reservoirs were studied but also the microscopic production laws of different occurrence states were studied. The results show that the pore distribution of Jilin shale is more concentrated than that of Qinghai shale. The oil of the two blocks mainly occurs in 0.01–10 ms pores, and the occurrence ratio of Jilin shale in the pores is higher, which is more than 90%. The oil production of the two blocks is mainly dominated by 0.01–10 ms pores, and the utilization efficiency contribution of these pores in Jilin shale is higher, accounting for about 80%. The utilization efficiency (UE) increases logarithmically with centrifugal force, and the growth rate of Jilin shale is greater than that of Qinghai shale. The proportion of free oil in Jilin block is less than that in Qinghai block. The shale oil in the two blocks is both at 15% final UE, and the UE of free oil in Jilin shale is about 9% and that of Qinghai shale is about 12%. The recoverability of Jilin shale is lower than that of Qinghai shale.

1. Introduction

Foreign research institutions and scholars have not yet formed a unified understanding of the definition of shale oil, and the definition and limit are different. The U.S. Energy Information Administration uses shale oil and tight oil as the same concept, and the shale oil in the United States is broadly defined as oil contained in low-porosity and low-permeability tight reservoirs. Domestic shale oil has a clear definition. Shale oil refers to the oil resources occurring in the organic-rich shale strata, in which the single-layer thickness of siltstone, fine sandstone, and carbonate rock in the source rock of organic-rich shale strata is no more than 5 m, and the cumulative thickness accounts for less than 30% of the total thickness of shale strata. There is no natural production capacity or capacity lower than the lower limit of industrial oil production, and special technological measures are needed to obtain industrial oil production [1–6]. At present, China's shale oil exploration and development is in the initial stage and has made breakthroughs in many typical blocks, such as Junggar, Ordos, Qaidam, and Sichuan, and the recoverability of shale oil in different regions varies. Shale oil exists in the organic-rich shale system in the adsorbed, free, and dissolved states. At present, the potential mobile and recoverable shale oil mainly exists in the adsorbed and free states. The adsorbed state mainly occurs on the surface of kerogen and clay particles, and the free state mainly occurs in matrix pores and microfractures. The accumulation and occurrence form of pores at different scales directly affect the recoverability of shale oil [7, 8]. Shale oil reservoirs have strong heterogeneity, small pore-throat structure, complex pore space distribution, organic matter structure, fluid phase, and oil and gas contents. Many traditional analysis methods for reservoir recoverability are not applicable. Previous studies have investigated the practicability from both geological and experimental perspectives. In terms of geology, Zhang et al. [9] analyzed the recoverability of shale oil from the perspective of formation energy through well-logging data combined with experimental analysis. According to the calculation formula of elastic recoverable reserves of the reservoir, the calculation formula of the elastic movable oil rate of shale oil was derived. Freedman et al. [10] provided a reservoir evaluation method to accurately determine the porosity and fluid volume of shale oil reservoir logging organic shale reservoirs. Jiang et al. [11] proposed the quantitative evaluation coefficient of oil and gas mobility in the "sweet spot" of shale oil geology by studying the quantitative evaluation method of oil and gas mobility in the "sweet spot" of shale oil geology through logging evaluation. Jin et al. [12] used electron beam charging effect, conductivity test, polar solvent extraction, heating, and other methods to carry out a systematic comparative evaluation of shale oil core samples in each block of the four major basins, including Junggar Basin, Bohai Bay Basin, Ordos Basin, and Songliao Basin, based on the same evaluation technical system and the same experimental test conditions. A systematic understanding of the recoverability of shale oil resources in each basin at the micro scale was formed. Shi et al. [13] used the pyrolysis method to evaluate the recoverability of Chang 7 shale oil. In terms of experiments, Jiang et al. [14] used nuclear magnetic resonance (NMR) technology combined with gradual centrifugation and gradual drying experiments to quantitatively evaluate the pore fluids of shale reservoirs and established the evaluation technology and method for identifying the types of pore fluids of shale reservoirs. Jiang et al. [15] studied the recoverability of shale oil by using NMR technology combined with centrifugal experiments and found that the mobile oil mainly occurred in large pores, and the bound oil was mainly in small pores. Mitchell [16], Yuan [17], Yang [18], and Sang [19] et al. used NMR technology to test the movable fluid of shale oil blocks in Jiyang Depression. Gong et al. [20] used NMR to establish a method to distinguish shale oil in organic matter and inorganic matter and to evaluate the mobility of shale oil in the Ordos Basin. In conclusion, for the recoverability evaluation of shale oil reservoir, the research from the perspective of geology is relatively perfect, and the research from the perspective of development is slightly weak. Based on this, as shown in Figure 1, in the premise of previous one-dimensional (1D) NMR analysis of pore utilization characteristics, this study introduced a two-dimensional (2D) NMR analysis of microscopic utilization laws in different occurrence states. A comparative study on the recoverability of shale oil reservoirs in Qinghai and Jilin was carried out to provide theoretical guidance for the effective development of typical shale oil blocks.

2. Experiment

2.1. Experimental Material. The typical shale oil rock cores selected in the experiment are from the Qinghai shale oil reservoir in Qaidam Basin and Jilin shale oil reservoir in Songliao Basin, and the geological characteristics of different blocks are different.

The shale reservoir in Qaidam Basin mainly develops Paleogene saline lacustrine shale. The "two-stage" continuous and efficient hydrocarbon generation of low-abundance shale in the saline lacustrine basin provides the basis for oil and gas enrichment. The high carbonate mineral content and low clay mineral content and the development degree of laminate structure lay the foundation for the reservoir reconstruction of the shale oil reservoir [21–24]. In addition, good crude oil quality, high free oil content, and high formation pressure and gas-oil ratio provide sufficient power for oil and gas flow. The exploration of oil and gas resources shows that it has a good resource prospect, with a geological resource of 21×10^8 t. It is difficult to construct because of the unique plateau and mountainous environment, low temperatures, hypoxia, and water scarcity. The huge thick deposition of over kilometers and the rugged surface determine the diversity of well site selection, well opening mode, and well type selection. The theory and technology of exploration and development of shale reservoir at home and abroad cannot be completely copied [25–27].

The shale reservoir in Songliao Basin is the first exploration of pure shale reservoir exploration and development in China. In 2019, the shale oil of Cretaceous Qingshankou Formation in Guye well obtained high-yield industrial oil flow, which realized the leap from oil generation to production of continental shale oil. Shale oil in Songliao Basin is significantly different from that in North American Marine shale oil and saline lake basin shale oil in terms of mineral composition, physical characteristics, oil content, and recoverability. Especially, its laminated structure and foliation structure are extremely developed. Therefore, the occurrence mechanism and transportation mechanism of shale oil in the pore-fracture system are relatively complex, and the exploration and development mode of shale oil in North America and other areas cannot be copied [28–30].

Table 1 shows the selected rock cores basic physical property parameters. The porosity of Qinghai shale oil rock cores ranges from 7.69% to 11.53%, with an average of 9.31%, and the permeability ranges from 0.281 to 0.425 mD, with an average of 0.336 mD. The porosity of Jilin shale oil rock cores is between 2.06% and 8.37%, with an average of 4.70%, and the permeability is between 0.014 and 0.038 mD, with an average of 0.029 mD. From the perspective of

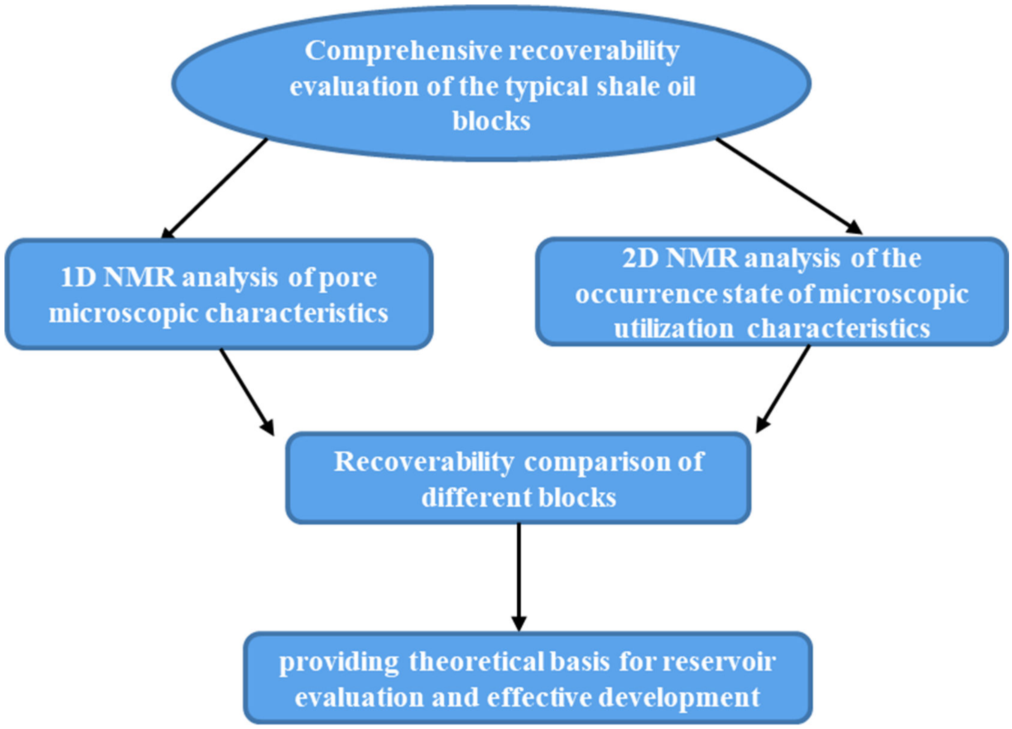

Figure 1: The flow chart of the research profile.

physical property parameters, the pore development degree of Qinghai shale oil core is better than that of Jilin shale oil core, and the flow capacity is also stronger.

2.2. Experimental Procedures.

(1) The rock cores were placed in a drying oven and dried at 110°C for 48 hours.

(2) The porosity and permeability of the cores were measured by helium and nitrogen, respectively.

(3) The rock cores were evacuated and pressurized to saturate the simulation oil for 2 days. The simulation oil was kerosene. The density at room temperature was 0.80 g/cm^3, and the surface tension of kerosene was 25.98 mN/m.

(4) The saturated oil rock cores were centrifuged at different speeds of 2500 R/min, 5000 R/min, 7500 R/min, and 10,000 R/min for an hour.

(5) 1D NMR and 2D NMR of rock cores at different stages were measured. Among them, No. 1 rock core was fractured during centrifugation at 10,000 R/min. Therefore, the data after centrifugation at this speed could not be obtained.

2.3. Experimental Method. NMR is a phenomenon in which certain nuclei with spin magnetic moments absorb electromagnetic waves of specific frequencies under the action of an external magnetic field, thereby changing the energy state [31–33]. When NMR technology is applied in petroleum exploration and development, physical and fluid parameters, such as reservoir permeability, porosity, oil saturation, and percentage of movable fluid, can be quickly obtained by testing rock samples with NMR [34, 35]. It provides an effective method and means for the division and evaluation of effective reservoirs. At present, there are many researches on 1D NMR evaluation method. Compared with 1D NMR evaluation method, 2D NMR evaluation method can not only quantitatively analyze the utilization characteristics of the reservoir but also quantitatively and intuitively analyze the microscopic utilization law of the reservoir occurrence state [36–39]. In this study, based on the previous 1D NMR evaluation method, the 2D NMR evaluation method was introduced to provide a theoretical basis for the recoverability evaluation of typical shale oil reservoirs. In this study, off-line NMR instruments were used. Recore-04 core NMR analyzer is an experimental instrument developed by the Institute of Flow Fluid Mechanics, Chinese Academy of Sciences. The main test parameters are below, echo interval time is 300 μm, echo number is 1024, scan times is 64, wait time is 3000 ms, and gain is 50.

3. Results and Discussions

3.1. Analysis of 1D NMR Results

3.1.1. Shale Oil Block in Qinghai. Figure 2 shows the NMR T_2 spectrum at different stages in Qinghai block. It can be seen from the figure that the signal of each relaxation interval is distributed, and the pore distribution of Qinghai

Table 1: Physical property parameters of rock cores.

No.	Block	Length/cm	Diameter/cm	Porosity/%	Permeability/mD
1	Qinghai	7.29	2.53	11.53	0.425
2	Qinghai	7.34	2.52	8.71	0.301
3	Qinghai	6.95	2.52	7.69	0.281
4	Jilin	7.86	2.57	2.06	0.014
5	Jilin	7.21	2.54	3.68	0.036
6	Jilin	7.10	2.51	8.37	0.038

shale is relatively uniform. Moreover, the NMR signal of each relaxation interval decreased with the increase in centrifugal speed. It can be seen from the figure that with the increase in centrifugal speed, the NMR T_2 spectrum gradually shifts to the left. According to the NMR surface relaxation principle, the stronger the interaction force between hydrogen-containing fluid and the pore-throat surface, the shorter the relaxation time, and vice versa. Compared with before displacement, the relaxation time after centrifugation becomes shorter when it moves to the left. Therefore, the interaction force between fluid and pore-throat surface becomes stronger with the increase of in centrifugal speed. That is, the fluid in the small pores is centrifuged to the throat surface.

The centrifugal speed was converted into centrifugal force, and the centrifugal forces corresponding to 2500 R/min, 5000 R/min, 7500 R/min, and 10,000 R/min were 0.375, 1.500, 3.375, and 6.000 MPa, respectively. Figure 3 shows the variation trend of the utilization efficiency (UE) in Qinghai shale reservoir with centrifugal force. It can be seen from the figure that the UE and centrifugal force show a logarithmic correlation. Because the larger the radius of the pore throat, the smaller the corresponding capillary resistance. So the crude oil in the large pore throat is first centrifuged and then the crude oil in the smaller pore throat is centrifuged to the surface of the rock sample during the centrifugation process, which means that the UE increases logarithmically with the centrifugal force. With the increase in centrifugal force, the UE increased, and the final UE of rock cores No. 1 to No. 3 is 16.7%, 14.3%, and 13.9%, respectively. The average UE is 15.0%. The higher the rock cores permeability, the greater the final UE. The 1D NMR results were quantitatively analyzed, and the results are shown in Table 2.

As can be seen from Table 2, the content proportion of oil in 0.01–10 ms pores of rock samples No. 1 to No. 3 is 76.16%, 85.58%, and 82.96%, respectively, with an average of about 81.57%. Therefore, oil mainly occurs in 0.01–10 ms pores. With the increase in centrifugal speed, the UE of pores with different relaxation intervals also increased. Finally, the UE of 0.01–1 ms pores of rock samples No. 1 to No. 3 increased by 1.052%, 6.900%, and 2.551%, respectively, with an average of 3.501%. And the UE of 1–10 ms pores increased by 5.579%, 3.299%, and 4.523%, with an average of 4.467%, that of 10–100 ms pores increased by 2.996%, 0.576%, and 2.697%, with an average of 2.090%. Finally, the UE of 100–1000 ms pores

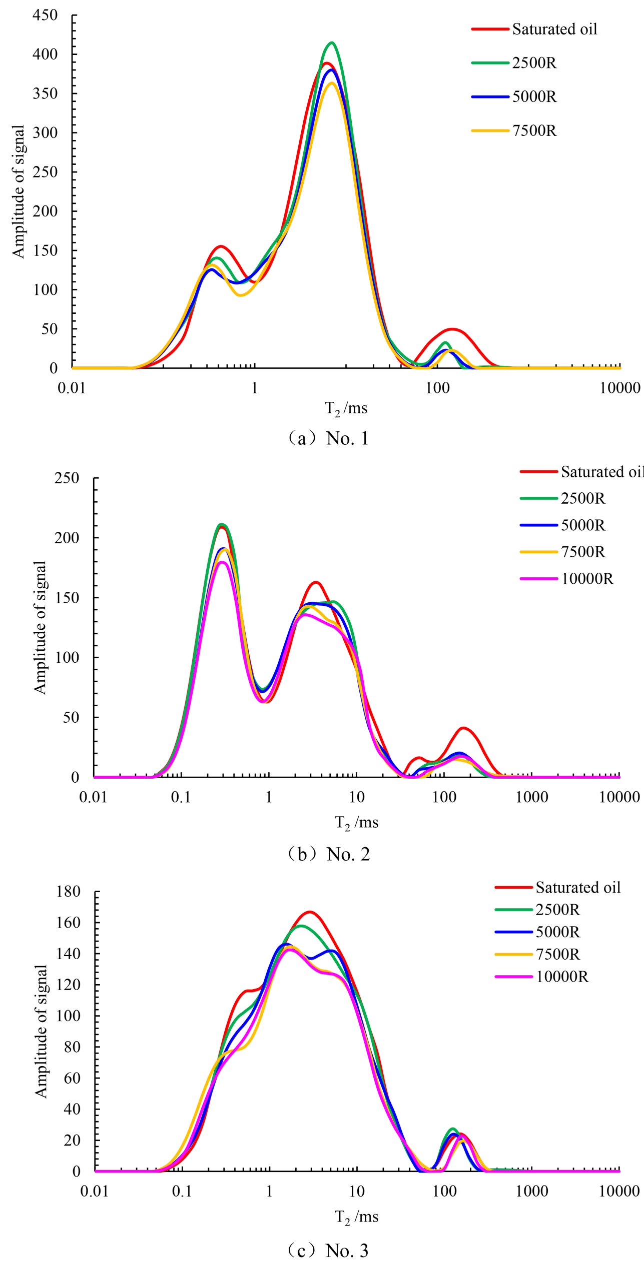

Figure 2: 1D NMR T_2 spectrum in Qinghai shale oil block.

increased by −0.08%, −0.108%, and 0.121%, respectively. It can be seen with the increase in centrifugal speed, the UE

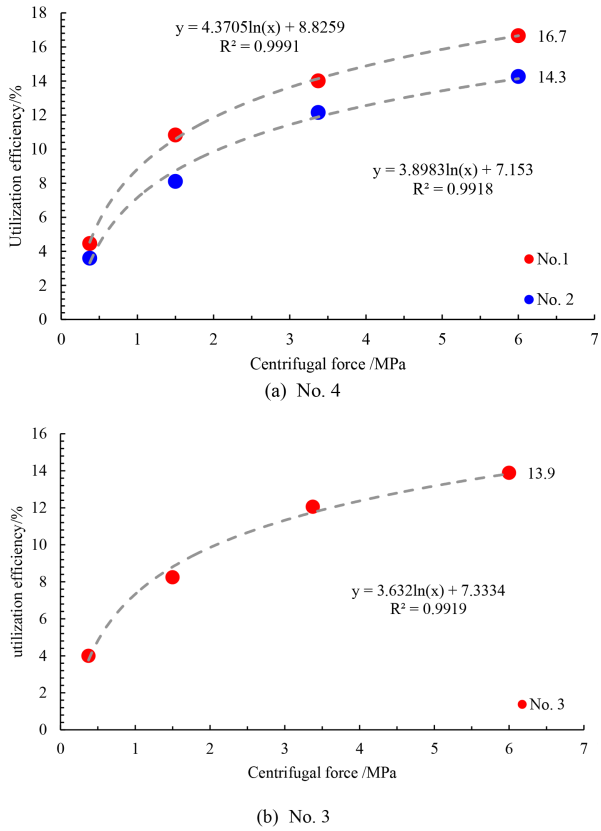

FIGURE 3: Variation trend of utilization efficiency with centrifugal force in Qinghai reservoir.

TABLE 2: One-dimensional NMR analysis in Qinghai reservoir.

No.	Occurrence proportion of relaxation time /%		Absolute utilization efficiency%			
			2500R	5000R	7500R	10,000R
1	0.01–1	21.979	0.051	0.957	1.103	/
	1–10	54.183	0.004	3.635	5.583	
	10–100	19.760	1.409	3.049	4.405	
	100–1000	4.078	2.991	3.182	2.911	
2	0.01–1	45.763	0.720	3.283	4.162	6.175
	1–10	39.816	1.171	0.727	1.395	2.128
	10–100	9.054	1.975	2.377	3.087	2.551
	100–1000	5.368	3.510	3.168	3.501	3.402
3	0.01–1	30.624	0.888	2.087	2.517	3.439
	1–10	52.333	2.329	4.217	6.585	6.852
	10–100	14.367	0.288	1.186	2.382	2.985
	100–1000	2.677	0.488	0.748	0.570	0.609

of 0.01–10 ms pores increased the most, followed by 10–100 ms, and UE of 100–1000 ms pores increased the least. The final UE of 0.01–10 ms pores of No. 1 to No. 3 rock samples is 6.69%, 8.30%, and 10.29%, with an average of 8.43%. And then the UE of 10–100 ms pores was 4.41%, 2.55%, and 2.99%, with an average of 3.32%. Besides, the UE of 100–1000 ms pores is 2.91%, 3.40%, and 0.61%, with an average of 2.31%. In conclusion, the utilization efficiency contribution (UEC) of 0.01–10 ms pore was the largest, accounting for about 60%, followed by 10–100 ms pores, accounting for about 24%. The UEC of 100–1000 ms pores was the smallest, accounting for about 16%.

3.1.2. Shale Oil Block in Jilin. Figure 4 shows the NMR T_2 spectrum at different stages in Jilin shale reservoir. It can be seen from the figure that compared with the Qinghai shale, Jilin shale has a more concentrated pore distribution. Among them, the pores of rock samples in Figure 4(a) are mainly distributed between 0.1 and 1 ms, and the pores in Figure 4(b) and 4(c) are mainly distributed between 1 and 10 ms. The pore sorting was worse, and the pores in the relaxation interval decreased centrally with the centrifugal speed. Compared with Figure 2, the shale oil in Qinghai belongs to an interbedded shale oil reservoir, which is fine sandstone, so it reflects some physical properties of a tight oil core. Jilin shale oil reservoir belongs to pure shale oil with weaker flow capacity, so it is different from Qinghai shale oil core.

Figure 5 shows the variation trend of UE with centrifugal force in Jilin shale block. It can be seen from the Figure 5 that, in general, with the increase in centrifugal force, the UE of Jilin shale increased at a higher rate than that of Qinghai shale. The final UE of No. 4 to No. 6 rock samples is 12.2%, 16.0%, and 16.6%, with an average of 15.0%, which is equivalent to that of Qinghai shale. The 1D NMR results of the shale in Jilin block after centrifugation were quantitatively analyzed, and the results are shown in Table 3.

As can be seen from Table 3, the content proportion of oil in 0.01–10 ms pores of No. 4 to No. 6 rock samples is 91.39%, 99.25%, and 94.85%, respectively, with an average of 95.16%. In general, the content proportion of oil in 0.01–10 ms pores is higher than that of

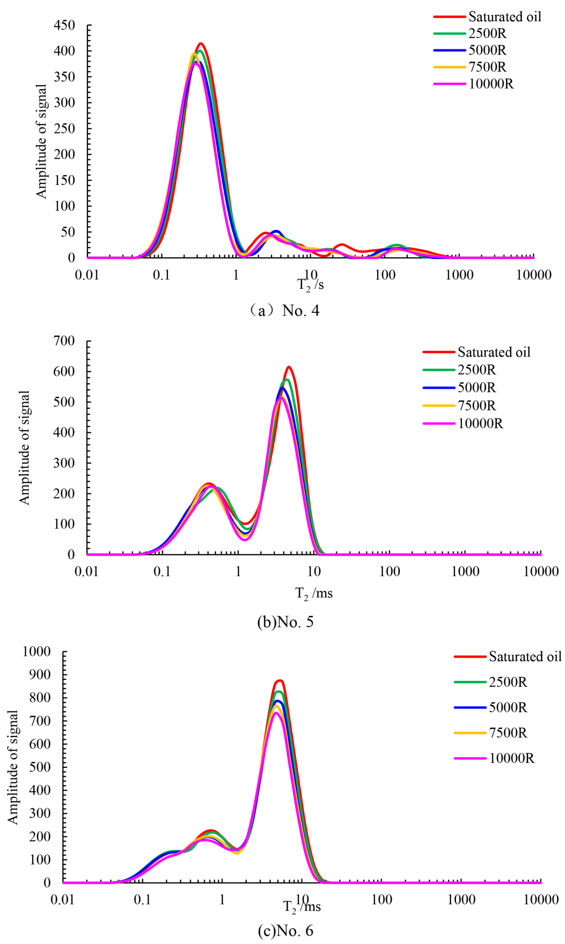

Figure 4: 1D NMR T_2 spectrum in Jilin shale oil block.

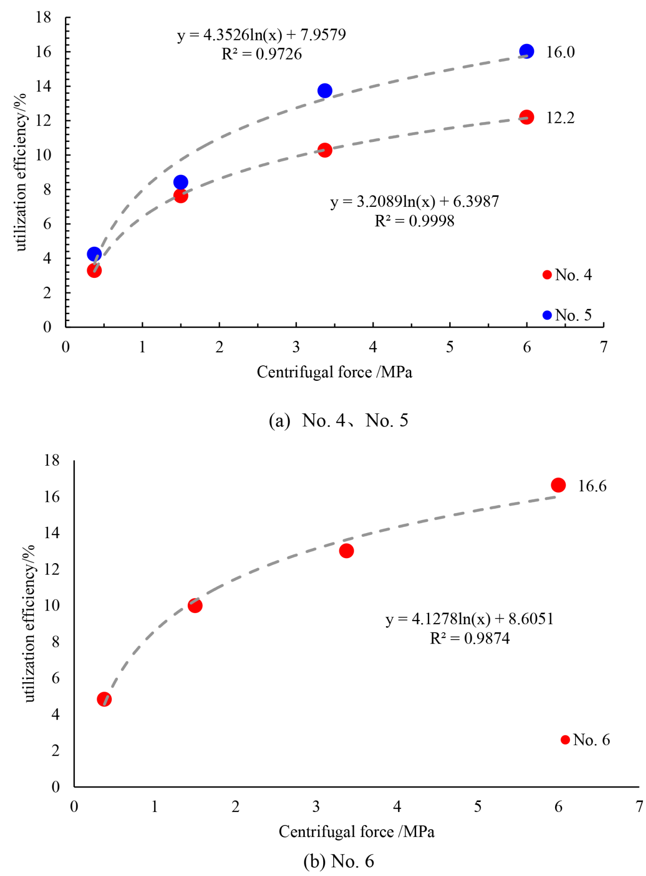

Figure 5: Variation trend of utilization efficiency with centrifugal force in Jilin reservoir.

Qinghai shale. With the increase in centrifugal speed, the UE of each relaxation interval also increased. Finally, the UE of 0.01–1 ms pores of No. 4 to No. 6 rock samples increased by 6.68%, 3.18%, and 3.25%, respectively, with an average of 4.37%. The UE of 1–10 ms pores increased by 1.11%, 8.07%, and 6.71%, respectively, with an average of 5.30%. Then the UE of 10–100 ms pores increased by 0.84%, 0.54%, and 1.84%, respectively, with an average of 1.07%. Finally, the UE of 100–1000 ms pores increased by 0.29%, 0%, and 0%, respectively. It can be seen that with the increase in centrifugal speed, the UE of 0.01–10 ms pores increased the most, followed by 10–100 ms pores, and that of 100–1000 ms pores is the smallest. The final UE of 0.01–1 ms pores of No. 4 to No. 6 rock samples was 6.57%, 3.88%, and 3.49%, with an average of 4.65%. The UE of 1–10 ms pores is 1.80%, 11.49%, and 10.47%, with an average of 7.92%. The UE of 10–100 ms pores is 2.59%, 0.66%, and 2.67%, with an average of 1.97%. The UE of 100–1000 ms pores is 1.25%, 0%, and 0%, with an average of 0.42%. In summary, the UEC of 0.01–10 ms pores in Jilin shale is about 84% and that of 10–100 ms pores is about 13%. The UEC of 100–1000 ms pores is about 3%.

Compared with the Qinghai shale oil rock cores, the occurrence proportion of oil in the 0.01–10 ms pores of Jilin shale is larger, the UE of oil in the 100–1000 ms pores is smaller, and the UEC of pores is more concentrated.

3.2. Analysis of 2D NMR Results

3.2.1. Shale Oil Block in Qinghai. Figure 6 shows 2D NMR images of Qinghai shale at different stages (the closer the color is to the warm color, the higher the fluid saturation). It can be seen from the figure that each occurrence state saturation begins to weaken as the centrifugal speed increases. The 2D NMR results were quantitatively analyzed and the results are shown in Table 4.

It can be seen from Table 4 that the proportion of free oil in rock samples No. 1 to No. 3 is 89.28%, 55.35%, and 88.46%, respectively, with an average of 77.70%. The proportion of adsorbed oil in rock samples No. 1 to No. 3 is 10.72%, 44.66%, and 11.54%, with an average of 22.30%. The occurrence state in Qinghai is mainly free oil. With the increase in centrifugal speed, the UE of each occurrence state increased. The free oil of rock samples No. 1 to No. 3 increased by 10.13%, 3.85%, and 8.25%, with an average of 7.41%, and the adsorbed oil increased by 1.34%, 6.73%, and 1.63%, with an average of 3.23%. In general, with the increase in centrifugal speed, the UE of the free state increased more. Finally, among the 15% UE of Qinghai

Table 3: One-dimensional NMR analysis of Jilin shales.

No.	Occurrence proportion of relaxation time/%		Absolute utilization efficiency/%			
			2500R	5000R	7500R	10,000R
4	0.01–1	81.988	0.118	2.873	5.488	6.566
	1–10	9.406	0.690	1.497	1.253	1.795
	10–100	4.986	1.75	1.933	2.358	2.590
	100–1000	3.621	0.96	1.328	1.182	1.250
5	0.01–1	33.556	0.694	1.011	2.867	3.878
	1–10	65.696	3.425	6.947	10.340	11.490
	10–100	0.749	0.121	0.461	0.530	0.657
	100–1000	0	0	0	0	0
6	0.01–1	25.531	0.238	1.173	2.319	3.491
	1–10	69.315	3.758	6.827	8.488	10.472
	10–100	5.154	0.834	1.990	2.201	2.672
	100–1000	0	0	0	0	0

shale oil, the UE of free state is about 12%, and the UE of adsorbed oil is about 3%.

3.2.2. Shale Oil Block in Jilin. Figure 7 shows the 2D NMR images of Jilin No. 3 shale, the quantitative analysis of the 2D NMR results was carried out, and the results are shown in Table 5.

As can be seen from Table 5, the proportion of free oil in Jilin rock samples No. 4 to No. 6 is 9.62%, 68.95%, and 76.92%, respectively, with an average of about 51.83%. The proportion of adsorbed oil is 90.38%, 31.05%, and 23.09%, respectively, with an average of about 48.17%. In general, the proportion of free oil in Jilin shale is smaller than that of Qinghai shale. With the increase in centrifugal speed, the UE of each occurrence state increased. The increase of free oil of rock samples No. 4 to No.6 is 1.01%, 8.51%, and 7.36%, with an average of 5.63%, and the increase of adsorbed oil is 8.13%, 3.80%, and 4.45%, with an average of 5.46%. The increase of each occurrence state is relatively uniform. In the final UE of about 15% in Jilin shale oil, the UE of free oil is about 9%, and the UE of adsorbed oil is about 6%. The UEC of adsorbed oil is higher than that of Qinghai shale, and the UEC of free oil is lower than that of Qinghai shale. Therefore, the mobility of Jilin shale oil is lower than that of Qinghai shale oil, which is consistent with the results of 1D NMR analysis. In the process of actual exploration and development, a larger centrifugal force or mining pressure is needed to extract the adsorbed oil.

4. Conclusions

From the perspective of exploration and development, based on the previous 1D NMR evaluation method, combined with the 2D NMR evaluation method, this research evaluates the recoverability of typical shale oil reservoirs through centrifugal physical simulation experiments. It would be better if a geological analysis of

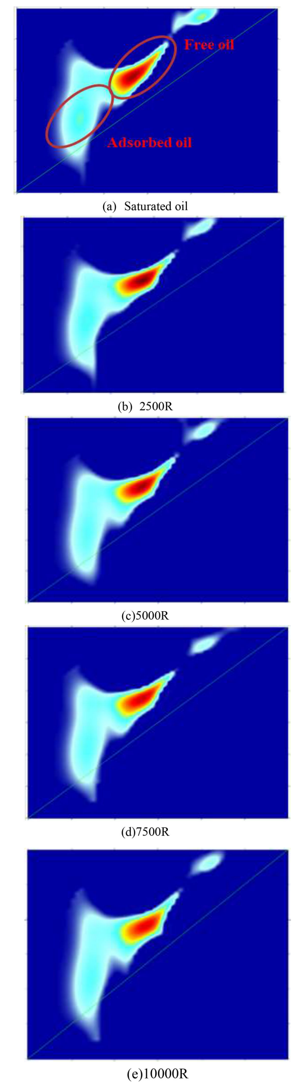

Figure 6: 2D NMR images of Qinghai No. 2 shale.

the core could be carried out to verify the experimental analysis. The following conclusions are obtained.

(1) The pore distribution of Qinghai shale is more uniform, and the pore distribution of Jilin shale is more concentrated. The oil of the two blocks mainly occurs in the 0.01–10 ms pores, and the content of Jilin shale oil in the pores is more. The UEC of the 0.01–10 ms pores of the two blocks is the highest. The UEC of the 0.01–10 ms pores in Qinghai block is about 60% and that in Jilin block is about 80%.

(2) The UE of the two blocks increased logarithmically with the centrifugal force, and the increase rate of Jilin shale was higher than Qinghai shale. Therefore, in the actual mining process, Jilin shale oil block needs a larger centrifugal force than Qinghai shale oil block..

(3) The free oil of the movable oil in Qinghai block accounts for about 70%, which is larger than that in

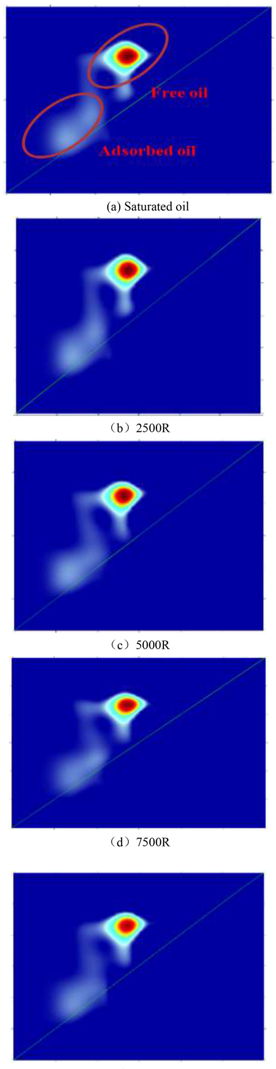

Figure 7: 2D NMR images of Jilin No. 6 shale.

Table 4: Two-dimensional NMR analysis of Qinghai shales.

No.		Proportion of occurrence/%	Absolute utilization efficiency/%			
			2500R	5000R	7500R	10,000R
1	Free oil	89.284	5.204	11.477	15.329	/
	Adsorbed oil	10.716	2.013	1.253	0.673	
2	Free oil	55.345	4.402	7.626	10.563	8.246
	Adsorbed oil	44.655	0.302	1.309	2.205	6.430
3	Free oil	88.462	3.706	7.928	11.953	11.962
	Adsorbed oil	11.538	0.287	0.310	0.049	1.923

Jilin block, and the adsorbed oil in Qinghai block is smaller than that in Jilin block. Under the same centrifugal force, the final UE of Qinghai shale oil and Jilin shale oil is about 15%. Among the UE of

Table 5: Two-dimensional NMR analysis of Jilin shales.

No.		Proportion of occurrence/%	Absolute utilization efficiency/%			
			2500R	5000R	7500R	10,000R
4	Free oil	9.621	0.067	0.580	0.911	1.079
	Adsorbed oil	90.379	0.793	3.876	9.588	8.919
5	Free oil	68.946	4.464	8.150	11.408	12.974
	Adsorbed oil	31.054	1.553	2.670	4.557	5.346
6	Free oil	76.915	4.782	9.159	10.140	12.138
	Adsorbed oil	23.085	0.047	0.832	2.868	4.497

15%, the UE of free oil in Qinghai shale is about 12% and that in Jilin shale is about 9%. The mobility of Jilin shale oil is lower than that of Qinghai shale oil.

Data Availability

The data for this study are available in this manuscript.

Conflicts of Interest

The authors declare that they have no conflict of interest.

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